

CRYSTAL STRUCTURE ANALYSIS OF K_3VF_6 COMPOUND

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Abstract

The object of research is the crystal structure of the α phase of K_3VF_6 compound. From the analysis of the literature it follows that there are three modifications of this compound – the α phase, the β phase and the γ phase. There are also several methods for preparing this compound. One of the problem areas is the existence of a large number of diffraction spectra obtained with the Bragg-Bertrand survey geometry in the PDF-2 database for 2004. There is also no complete data on the crystal structure of this compound. A structural model is proposed for the diffraction spectrum of the compound under the number 00-037-0738.

The study used the PDF-2 database for 2004. As well as the HighScorePlus program, which allows to refine the microstructural parameters of the structural model by the Rietveld method.

As a result, it is found that this diffraction spectrum of the compound under study can correspond to the following structural model: tetragonal system with lattice periods $a=12.3524(4)$ Å; $b=12.3524(4)$ Å; $c=16.7447(7)$ Å. A possible spatial symmetry group is $I4_1/a$ (88).

Analyzing the obtained results, it can be assumed that the studied structure of the α phase of the compound crystallizes in its own structural type.

Keywords: X-ray diffraction analysis, Bragg-Bertrand survey geometry, Rietveld method, crystal structure, K_3VF_6 composition.

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1. Introduction

The synthesis, crystalline structure and properties of mixed secondary metals are becoming an area of interest for scientists of material science for their isoelectronic and isostructural nature with their oxide counterparts [1].

Unlike oxides, the synthesis of mixed metal fluorides is limited due to the complexity that occurs during its synthesis, and therefore many interesting properties inherent in these metal fluorides are intensively studied [1, 2].

The general methods adopted for the synthesis are high temperature solid-state synthesis, high hydrothermal pressure (100 MPa), sol-gel synthesis and a solution based on chemical synthesis.

K_3VF_6 compound can be obtained by light synthesis at room temperature for V (III) and potassium-containing KF secondarily.

The non-hygroscopic nature of K_3VF_6 makes it an ideal electrolyte component for electrorefining or electrostriction of vanadium compared to other candidates such as VCl_2 and VCl_3 , as well as in electrochemical cells [3, 4].

Therefore, investigation of the crystal structure of this compound remains relevant.

The object of research is the crystal structure of the α phase of K_3VF_6 compound.

At the same time, it follows from the literature that there are six diffraction spectra of the indicated compound obtained by X-ray powder diffraction (Bragg-Bertrand method) of different quality.

The aim of this research is to propose a structural model for the diffraction spectrum of the K_3VF_6 compound under the number 00-037-0738 in the database PDF-2 for 2004.

2. Materials and methods

To achieve this aim it is necessary to solve the following objectives:

- to index the diffraction spectrum of the compound at number 3 (according to **Table 1**);
- to determine the lattice and syngony periods;

- to choose a spatial symmetry group for a given spectrum of the compound;
- to propose a structural model for a given spectrum of the compound;
- to refine the microstructural parameters for the selected model by the Rietveld method.

Since part of KHF_2 in the mixture decomposes without reaction with V_2O_3 , the excess KHF_2 necessary for the preparation of K_3VF_6 compound. In a 300-gram portion of each mixture in molar ratios of 1:6, 1:6.5, 1:7, 1:8, 1:9, 1:10, 1:11 and 1: 12 were heated at 600 °C in a stream argon (200 ml/min.) using certain equipment described in detail in [5]. After cooling, the reaction products were washed with water, and the residue was examined with the naked eye and also by X-ray diffraction. Vanadium (III) oxide is black or brown, and K_3VF_6 is bright green. When the reaction product is washed with water, the remaining parts of V_2O_3 can be well separated from K_3VF_6 in water. Mixtures in molar ratios of 1:7–1:12 gave K_3VF_6 pure [5], **Table 1**.

Table 1Details of the K_3VF_6 crystal structure

| No. | Compound | Syngony, SSG | Lattice periods, Å° | Card No. in the pdf-2 base | Diffraction spectrum quality |
|-----|-------------------------|---------------|--|----------------------------|------------------------------|
| 1 | K_3VF_6 | Cubic, F | $a=8.7$ $b=8.7$ $c=8.7$ | 00-022-0868 | Indexed [6] |
| 2 | K_3VF_6 | Not known | Lattice periods are not defined | 00-028-0841 | Low spectrum accuracy [7] |
| 3 | K_3VF_6 | Monoclinic | $a=12.1120$ $b=17.4740$ $c=12.0190$ $\beta=92.59$ | 00-037-0738 | Highest Spectrum Quality [8] |
| 4 | K_3VF_6 | Monoclinic | $a=17.136$ $b=12.368$ $c=5.479$ $\beta=91.485$ | 00-037-0739 | Indexed [8] |
| 5 | K_3VF_6 | Tetragonal, I | $a=13.708$ $b=13.708$ $c=8.752$ | 00-038-0541 | Indexed [8] |
| 6 | K_3VF_6 | Tetragonal, I | $a=13.570$ $b=13.570$ $c=8.671$ | 00-038-0542 | Indexed [8] |

According to [8], the diffraction spectrum 00-037-0738 in the PDF-2 database of K_3VF_6 compound obtained at 298 0K as α phase, numbered 00-037-0739 at 453 K, as β phase, and are indexed in monoclinic syngony, and 00-038-0541 γ phase is formed at 483 K, and is indexed in tetragonal syngony.

An Internet source [13] indicates that the structure of the 3 phase of K_3VF_6 belongs to the structural type $[\text{NH}_4]_3\text{FeF}_6$ of the space group Fm-3m (225).

Spectrum number 3 in the Table 1, in my opinion, deserves special attention due to its high quality of shooting (as indicated in the card of this spectrum given in the PDF-2 database for 2004), as well as the lack of literature data on a detailed study of the structure of K_3VF_6 compound. According to [8], this diffraction spectrum is indexed in monoclinic syngony (**Fig. 1**).

As a result of the research, it is found that this system is a simple quasibinary system and for KCl-K it has a eutectic temperature of 604 °C with a composition of 55 mol % KCl, and for KCl- K_3VF_6 the eutectic point is at 741 °C and 7.5 mol % K_3VF_6 . It is also found that KF- K_3VF_6 is a simple eutectic type at a temperature of 821 °C and 7.5 mol % K_3VF_6 . The system and the

triple eutectic point at 594 °C at the point KCl 54.8, KF 44, K_3VF_6 1.2 mol.%. Thus, the eutectic points have a much higher temperature than the temperature of formation of the K_3VF_6 phases according to [8].

Also in [10], the importance of F – as a stabilizing ligand for V (III) and Mn (III) is emphasized, both in aqueous solution and in solid compounds. Many fluorine and mixed fluorine complexes have been synthesized from an aqueous solution. $VF(C_5H_7O_2)_2$, Na_3VF_6 and K_3VF_6 compounds are obtained on the basis of electron transfer processes, including vanadium (V) and harmful reducing agents.

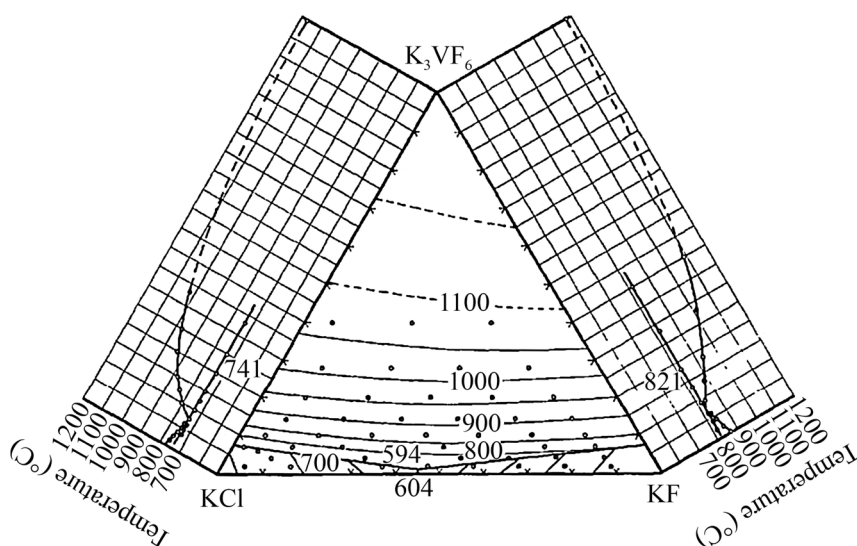


Fig. 1. System status diagram KCl-KF- K_3VF_6

In [11], the creation of the K_2VF_6 compound by fluorination with K_3VF_6 is noted. However, there is no information on the crystal structure of the input compound.

According to [12], K_3VF_6 is obtained by the interaction of powder V with KHF_2 . Green crystalline K_3VF_6 is separated by sublimation from Reacting V metal and from KF, which is formed in the reaction and is detected by chemical analysis. Previously, K_3VF_6 is prepared by reacting VF_3 with KF. Vanadium powder is heated with excess KHF_2 in a platinum crucible to 600 °C, and slowly rises to 800 °C. The product, a pale green melt, is then heated for a long time at 1200 °C in a graphite crucible. Bright green crystals condense in the upper parts of the crucible. All excess KF and a small portion of the green product crystallized on colder thermal screens.

The diffraction spectrum for the study is generated using the HighScorePlus 3.0 program [14] and the attached pdf-2 database for 2004 in the UDF format.

An analysis of the proposed structural model of this spectrum is also performed using the HighScorePlus 3.0 program by the Rietveld method. Obtaining initial data for studying the crystal structure of the compound, exclusively data taken from the PDF-2 database for 2004 are used.

3. Research results

The diffraction spectrum of the compound is indexed in tetragonal system with lattice periods $a=12.3524(4)$ Å; $b=12.3524(4)$ Å; $c=16.7447(7)$ Å. The space group of symmetry I 41/a (88) is possible.

An analysis of the proposed structural model of this spectrum is also performed using the HighScorePlus 3.0 program by the Rietveld method.

The correct system of points and their coordinates for this spectrum are specified in Table 2.

Table 2
K₃VF₆ microstructural parameters

| Atom | The correct point system | x | y | z | Position fill ratio | U_{iso}^a |
|------|--------------------------|-----------|-----------|-----------|---------------------|-------------|
| K1 | 16f | 0.2313(8) | 0.7620(8) | 0.4875(5) | 1.000000 | 0.2(2) |
| K2 | 16f | 0.239(1) | 0.016(1) | 0.1285(9) | 1.000000 | 4.0(5) |
| K3 | 16f | 0.045(2) | 0.061(3) | 0.146(2) | 0.500000 | 10(1) |
| K4 | 16f | 0.149(2) | 0.658(2) | 0.509(1) | 0.500000 | 0(1) |
| V1 | 8e | 0.000000 | 0.250000 | 0.3777(8) | 1.000000 | 5(1) |
| V2 | 4b | 0.000000 | 0.250000 | 0.625000 | 1.000000 | 10(1) |
| V3 | 4a | 0.000000 | 0.250000 | 0.125000 | 1.000000 | 8(1) |
| F1 | 16f | 0.620(2) | −0.910(2) | −0.291(1) | 1.000000 | 0(1) |
| F2 | 8d | 0.000000 | 0.000000 | 0.500000 | 1.0(2) | 0(1) |
| F3 | 16f | 0.123(2) | 0.378(2) | 0.258(1) | 1.000000 | 0(1) |
| F4 | 16f | 0.311(3) | 0.666(2) | 0.400(2) | 1.000000 | 10(1) |
| F5 | 8e | 0.000000 | 0.250000 | −0.006(7) | 0.341316 | 10(6) |
| F6 | 16f | 0.252(2) | 0.530(3) | 0.485(2) | 1.000000 | 8(1) |
| F7 | 16f | 0.090(3) | 0.159(2) | 0.259(2) | 1.000000 | 7(1) |
| F8 | 8e | 0.000000 | 0.250000 | 0.689(4) | 0.658684 | 10(2) |

The values of interplanar distances and intensities observed and calculated by this model are given in **Table 3**.

Table 3
The values of interplanar distances and intensities, which are observed and calculated

| d_{cal} (Å°) | d_{obs} (Å°) | I_{cal} | I_{obs} | H | K | L | d_{cal} (Å°) | d_{obs} (Å°) | I_{cal} | I_{obs} | H | K | L |
|----------------|----------------|-----------|-----------|-----|-----|-----|----------------|----------------|-----------|-----------|-----|-----|-----|
| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 |
| 9.93 | — | 1.20 | — | 0 | 1 | 1 | 1.58 | — | 1.43 | — | 4 | 1 | 9 |
| 6.17 | — | 0.54 | — | 0 | 2 | 0 | 1.57 | — | 1.12 | — | 5 | 6 | 1 |
| 6.04 | — | 0.23 | — | 1 | 1 | 2 | 1.57 | — | 0.09 | — | 6 | 5 | 1 |
| 5.24 | 5.24 | 2.31 | 0.93 | 1 | 2 | 1 | 1.56 | — | 1.07 | — | 2 | 2 | 10 |
| 5.24 | — | 0.51 | — | 2 | 1 | 1 | 1.56 | — | 0.21 | — | 0 | 7 | 5 |
| 5.08 | — | 0.82 | — | 0 | 1 | 3 | 1.54 | 1.54 | 0.38 | 6.21 | 1 | 6 | 7 |
| 4.96 | 4.96 | 90.8 | 80.9 | 0 | 2 | 2 | 1.54 | — | 0.01 | — | 6 | 1 | 7 |
| 4.36 | 4.36 | 24.3 | 24.2 | 2 | 2 | 0 | 1.54 | — | 5.94 | — | 0 | 8 | 0 |
| 4.18 | 4.18 | 3.77 | 5.09 | 0 | 0 | 4 | 1.53 | — | 0.08 | — | 1 | 3 | 10 |
| 3.99 | 4.00 | 1.64 | 1.09 | 0 | 3 | 1 | 1.53 | — | 0.00 | — | 3 | 1 | 10 |
| 3.92 | 3.92 | 0.18 | 1.09 | 1 | 2 | 3 | 1.52 | 1.52 | 0.22 | 1.05 | 1 | 8 | 1 |
| 3.92 | — | 0.89 | — | 2 | 1 | 3 | 1.52 | — | 0.02 | — | 8 | 1 | 1 |
| 3.87 | — | 0.04 | — | 2 | 2 | 2 | 1.52 | — | 0.26 | — | 4 | 7 | 1 |
| 3.77 | 3.78 | 2.64 | 2.18 | 1 | 1 | 4 | 1.52 | — | 0.86 | — | 7 | 4 | 1 |
| 3.53 | 3.53 | 0.40 | 1.11 | 1 | 3 | 2 | 1.52 | — | 0.00 | — | 5 | 6 | 3 |
| 3.53 | — | 2.81 | — | 3 | 1 | 2 | 1.52 | — | 0.24 | — | 6 | 5 | 3 |
| 3.46 | 3.46 | 1.90 | 1.08 | 0 | 2 | 4 | 1.51 | — | 0.03 | — | 0 | 8 | 2 |
| 3.35 | 3.35 | 2.37 | 4.17 | 2 | 3 | 1 | 1.51 | — | 0.01 | — | 2 | 7 | 5 |
| 3.35 | — | 1.95 | — | 3 | 2 | 1 | 1.51 | — | 0.18 | — | 7 | 2 | 5 |

Continuation of Table 3

| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 |
|------|------|------|------|---|---|---|------|------|------|------|----|----|----|
| 3.31 | 3.31 | 8.97 | 7.26 | 0 | 3 | 3 | 1.51 | — | 0.05 | — | 3 | 7 | 4 |
| 3.23 | 3.23 | 2.13 | 2.05 | 0 | 1 | 5 | 1.51 | — | 0.09 | — | 7 | 3 | 4 |
| 3.08 | 3.08 | 41.2 | 33.6 | 0 | 4 | 0 | 1.51 | 1.51 | 11.8 | 10.3 | 4 | 4 | 8 |
| 3.02 | 3.02 | 100. | 100. | 2 | 2 | 4 | 1.51 | — | 0.14 | — | 0 | 1 | 11 |
| 2.94 | 2.94 | 0.13 | 1.98 | 1 | 4 | 1 | 1.50 | — | 0.14 | — | 4 | 5 | 7 |
| 2.94 | — | 4.21 | — | 4 | 1 | 1 | 1.50 | — | 0.07 | — | 5 | 4 | 7 |
| 2.91 | 2.91 | 0.62 | 1.99 | 3 | 2 | 3 | 1.49 | — | 0.05 | — | 2 | 8 | 0 |
| 2.91 | — | 2.84 | — | 2 | 3 | 3 | 1.49 | — | 0.20 | — | 8 | 2 | 0 |
| 2.89 | — | 0.08 | — | 0 | 4 | 2 | 1.48 | — | 0.17 | — | 3 | 5 | 8 |
| 2.86 | 2.87 | 0.32 | 2.19 | 1 | 2 | 5 | 1.48 | — | 0.43 | — | 5 | 3 | 8 |
| 2.86 | — | 3.61 | — | 2 | 1 | 5 | 1.48 | — | 0.02 | — | 0 | 5 | 9 |
| 2.85 | — | 0.08 | — | 1 | 3 | 4 | 1.48 | — | 0.26 | — | 3 | 4 | 9 |
| 2.85 | — | 0.00 | — | 3 | 1 | 4 | 1.48 | — | 0.62 | — | 4 | 3 | 9 |
| 2.76 | 2.76 | 1.01 | 2.19 | 2 | 4 | 0 | 1.48 | — | 0.00 | — | 1 | 7 | 6 |
| 2.76 | — | 1.52 | — | 4 | 2 | 0 | 1.48 | — | 0.04 | — | 5 | 5 | 6 |
| 2.74 | — | 2.06 | — | 3 | 3 | 2 | 1.48 | — | 0.26 | — | 7 | 1 | 6 |
| 2.65 | — | 1.02 | — | 1 | 1 | 6 | 1.47 | 1.47 | 0.37 | 2.08 | 1 | 8 | 3 |
| 2.63 | 2.63 | 1.66 | 2.17 | 1 | 4 | 3 | 1.47 | — | 0.38 | — | 4 | 7 | 3 |
| 2.63 | — | 0.45 | — | 4 | 1 | 3 | 1.47 | — | 0.40 | — | 7 | 4 | 3 |
| 2.62 | — | 1.27 | — | 2 | 4 | 2 | 1.47 | — | 0.98 | — | 8 | 1 | 3 |
| 2.62 | — | 2.18 | — | 4 | 2 | 2 | 1.47 | — | 0.30 | — | 2 | 8 | 2 |
| 2.59 | 2.59 | 1.51 | 2.18 | 0 | 3 | 5 | 1.47 | — | 0.28 | — | 8 | 2 | 2 |
| 2.54 | — | 2.66 | — | 0 | 2 | 6 | 1.47 | — | 0.01 | — | 0 | 4 | 10 |
| 2.48 | 2.48 | 25.5 | 21.8 | 0 | 4 | 4 | 1.46 | — | 0.12 | — | 0 | 6 | 8 |
| 2.44 | 2.45 | 0.00 | 11.4 | 0 | 5 | 1 | 1.46 | — | 0.01 | — | 2 | 1 | 11 |
| 2.44 | — | 4.45 | — | 3 | 4 | 1 | 1.46 | — | 0.37 | — | 1 | 2 | 11 |
| 2.44 | — | 0.93 | — | 4 | 3 | 1 | 1.45 | 1.45 | 1.01 | 2.02 | 4 | 6 | 6 |
| 2.39 | 2.39 | 2.92 | 10.4 | 0 | 2 | 5 | 1.45 | — | 0.88 | — | 6 | 4 | 6 |
| 2.39 | — | 4.84 | — | 2 | 3 | 5 | 1.45 | — | 0.27 | — | 3 | 6 | 7 |
| 2.38 | — | 0.13 | — | 3 | 3 | 4 | 1.45 | — | 0.00 | — | 6 | 3 | 7 |
| 2.35 | — | 5.72 | — | 2 | 2 | 6 | 1.45 | 1.45 | 0.01 | 3.06 | 6 | 6 | 0 |
| 2.34 | 2.34 | 1.08 | 9.49 | | 1 | 7 | 1.45 | — | 0.58 | — | 3 | 3 | 10 |
| 2.32 | 2.32 | 1.26 | 2.09 | 1 | 5 | 2 | 1.44 | 1.44 | 0.61 | 2.03 | 0 | 8 | 4 |
| 2.32 | — | 0.09 | — | 5 | 1 | 2 | 1.44 | — | 0.08 | — | 2 | 5 | 9 |
| 2.30 | 2.30 | 7.10 | 5.27 | 2 | 4 | 4 | 1.44 | — | 0.09 | — | 5 | 2 | 9 |
| 2.30 | — | 0.04 | — | 4 | 2 | 4 | 1.44 | — | 0.01 | — | 3 | 8 | 1 |
| 2.27 | 2.27 | 1.99 | 7.31 | 5 | 2 | 1 | 1.44 | — | 0.03 | — | 8 | 3 | 1 |
| 2.27 | — | 0.31 | — | 2 | 5 | 1 | 1.43 | 1.43 | 0.01 | 3.04 | 6 | 6 | 2 |
| 2.27 | — | 2.10 | — | 1 | 3 | 6 | 1.43 | — | 0.06 | — | 2 | 4 | 10 |
| 2.27 | — | 2.20 | — | 3 | 1 | 6 | 1.43 | — | 0.31 | — | 4 | 2 | 10 |
| 2.25 | 2.25 | 2.40 | 8.44 | 0 | 5 | 3 | 1.42 | — | 0.01 | — | 5 | 6 | 5 |
| 2.25 | — | 1.04 | — | 3 | 4 | 3 | 1.42 | — | 0.15 | — | 6 | 5 | 5 |
| 2.25 | — | 6.72 | — | 4 | 3 | 3 | 1.42 | 1.42 | 0.15 | 3.04 | 2 | 6 | 8 |
| 2.23 | 2.23 | 4.73 | 6.19 | 1 | 4 | 5 | 1.42 | — | 0.17 | — | 6 | 2 | 8 |

Continuation of Table 3

| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 |
|------|------|------|------|---|---|---|------|------|------|------|----|----|----|
| 2.23 | – | 1.14 | – | 4 | 1 | 5 | 1.42 | – | 0.01 | – | 0 | 3 | 11 |
| 2.19 | – | 0.12 | – | 1 | 2 | 7 | 1.41 | – | 0.02 | – | 0 | 7 | 7 |
| 2.19 | – | 0.22 | – | 2 | 1 | 7 | 1.41 | 1.41 | 0.12 | 1.02 | 5 | 7 | 2 |
| 2.18 | 2.18 | 63.5 | 54.7 | 4 | 4 | 0 | 1.41 | – | 0.00 | – | 7 | 5 | 2 |
| 2.12 | – | 0.45 | – | 2 | 5 | 3 | 1.41 | 1.41 | 0.08 | 1.02 | 2 | 8 | 4 |
| 2.12 | – | 0.01 | – | 5 | 2 | 3 | 1.41 | – | 0.50 | – | 8 | 2 | 4 |
| 2.11 | 2.11 | 0.05 | 2.09 | 4 | 4 | 2 | 1.40 | – | 0.02 | – | 3 | 7 | 6 |
| 2.09 | 2.09 | 0.79 | 28.2 | 1 | 5 | 4 | 1.40 | – | 0.00 | – | 7 | 3 | 6 |
| 2.09 | – | 0.07 | – | 5 | 1 | 4 | 1.39 | – | 0.07 | – | 3 | 8 | 3 |
| 2.09 | – | 25.0 | – | 0 | 0 | 8 | 1.39 | – | 0.24 | – | 8 | 3 | 3 |
| 2.07 | – | 0.01 | – | 0 | 4 | 6 | 1.39 | – | 0.02 | – | 0 | 0 | 12 |
| 2.06 | – | 1.25 | – | 0 | 3 | 7 | 1.39 | 1.39 | 0.25 | 1.02 | 1 | 8 | 5 |
| 2.05 | – | 0.01 | – | 0 | 6 | 0 | 1.39 | – | 0.77 | 4.10 | 4 | 7 | 5 |
| 2.05 | – | 0.93 | – | 3 | 5 | 2 | 1.39 | – | 0.25 | 3.07 | 7 | 4 | 5 |
| 2.05 | – | 0.07 | – | 5 | 3 | 2 | 1.39 | – | 0.09 | 2.05 | 8 | 1 | 5 |
| 2.03 | 2.03 | 0.63 | 1.09 | 1 | 1 | 8 | 1.39 | – | 0.03 | – | 2 | 3 | 11 |
| 2.01 | 2.01 | 0.67 | 2.16 | 1 | 6 | 1 | 1.39 | – | 0.11 | – | 3 | 2 | 11 |
| 2.01 | – | 0.19 | – | 6 | 1 | 1 | 1.38 | – | 0.24 | – | 2 | 7 | 7 |
| 2.01 | – | 0.58 | – | 3 | 3 | 6 | 1.38 | – | 0.18 | – | 7 | 2 | 7 |
| 1.99 | – | 0.11 | – | 0 | 6 | 2 | 1.38 | 1.38 | 1.00 | 4.10 | 4 | 8 | 0 |
| 1.98 | 1.98 | 2.14 | 18.8 | 0 | 5 | 5 | 1.38 | – | 0.86 | – | 8 | 4 | 0 |
| 1.98 | – | 1.45 | – | 3 | 4 | 5 | 1.37 | – | 0.06 | – | 1 | 1 | 12 |
| 1.98 | – | 11.8 | – | 4 | 3 | 5 | 1.37 | – | 0.39 | – | 1 | 5 | 10 |
| 1.98 | – | 0.02 | – | 0 | 2 | 8 | 1.37 | – | 0.01 | – | 5 | 1 | 10 |
| 1.96 | 1.96 | 0.30 | 2.15 | 2 | 4 | 6 | 1.37 | 1.37 | 3.50 | 3.07 | 6 | 6 | 4 |
| 1.96 | – | 1.50 | – | 4 | 2 | 6 | 1.37 | – | 0.23 | – | 1 | 6 | 9 |
| 1.96 | – | 0.06 | – | 2 | 3 | 7 | 1.37 | – | 0.32 | – | 6 | 1 | 9 |
| 1.96 | – | 1.18 | – | 3 | 2 | 7 | 1.36 | – | 0.03 | – | 0 | 9 | 1 |
| 1.95 | 1.95 | 1.29 | 2.12 | 2 | 6 | 0 | 1.36 | – | 0.36 | – | 4 | 8 | 2 |
| 1.95 | – | 3.83 | – | 6 | 2 | 0 | 1.36 | – | 0.29 | – | 8 | 4 | 2 |
| 1.93 | 1.93 | 3.10 | 3.18 | 4 | 4 | 4 | 1.36 | – | 0.03 | – | 0 | 2 | 12 |
| 1.91 | – | 0.89 | – | 4 | 5 | 1 | 1.35 | – | 0.00 | – | 5 | 7 | 4 |
| 1.91 | – | 0.29 | – | 5 | 4 | 1 | 1.35 | – | 0.00 | – | 7 | 5 | 4 |
| 1.90 | 1.90 | 0.00 | 2.08 | 1 | 6 | 3 | 1.35 | – | 0.00 | – | 1 | 4 | 11 |
| 1.90 | – | 0.18 | – | 6 | 1 | 3 | 1.35 | – | 0.01 | – | 4 | 1 | 11 |
| 1.90 | – | 0.76 | – | 2 | 6 | 2 | 1.35 | – | 0.01 | – | 0 | 8 | 6 |
| 1.90 | – | 2.04 | – | 6 | 2 | 2 | 1.34 | – | 0.01 | – | 1 | 9 | 2 |
| 1.89 | 1.89 | 0.04 | 3.15 | 5 | 2 | 5 | 1.34 | – | 0.02 | – | 9 | 1 | 2 |
| 1.89 | – | 1.26 | – | 2 | 5 | 5 | 1.34 | – | 0.26 | – | 1 | 7 | 8 |
| 1.88 | – | 0.90 | – | 3 | 5 | 4 | 1.34 | – | 0.01 | – | 7 | 1 | 8 |
| 1.88 | – | 1.12 | – | 5 | 3 | 4 | 1.34 | – | 0.06 | – | 5 | 5 | 8 |
| 1.88 | – | 1.19 | – | 2 | 2 | 8 | 1.33 | – | 0.04 | – | 4 | 5 | 9 |
| 1.86 | 1.86 | 0.03 | 1.10 | 1 | 4 | 7 | 1.33 | – | 0.39 | – | 5 | 4 | 9 |
| 1.86 | – | 0.39 | – | 4 | 1 | 7 | 1.33 | 1.33 | 0.58 | 2.05 | 2 | 9 | 1 |

Continuation of Table 3

| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 |
|------|------|------|------|---|---|---|------|---|------|----|----|----|----|
| 1.84 | 1.84 | 0.58 | 2.12 | 0 | 6 | 4 | 1.33 | — | 0.05 | — | 6 | 7 | 1 |
| 1.84 | — | 0.26 | — | 1 | 3 | 8 | 1.33 | — | 0.48 | — | 7 | 6 | 1 |
| 1.84 | — | 0.58 | — | 3 | 1 | 8 | 1.33 | — | 1.01 | — | 9 | 2 | 1 |
| 1.83 | — | 0.01 | — | 0 | 1 | 9 | 1.33 | — | 0.09 | — | 0 | 9 | 3 |
| 1.83 | — | 0.85 | — | 3 | 6 | 1 | 1.32 | — | 2.35 | — | 2 | 2 | 12 |
| 1.83 | — | 0.00 | — | 6 | 3 | 1 | 1.32 | — | 0.00 | — | 4 | 4 | 10 |
| 1.82 | 1.82 | 1.00 | 2.13 | 1 | 5 | 6 | 1.32 | — | 0.00 | — | 3 | 8 | 5 |
| 1.82 | — | 0.61 | — | 5 | 1 | 6 | 1.32 | — | 0.02 | — | 8 | 3 | 5 |
| 1.82 | — | 0.02 | — | 4 | 5 | 3 | 1.32 | — | 0.06 | — | 4 | 6 | 8 |
| 1.82 | — | 1.22 | — | 5 | 4 | 3 | 1.32 | — | 0.31 | — | 6 | 4 | 8 |
| 1.76 | 1.76 | 18.1 | 25.9 | 2 | 6 | 4 | 1.31 | — | 0.23 | — | 2 | 8 | 6 |
| 1.76 | — | 14.6 | — | 6 | 2 | 4 | 1.31 | — | 0.12 | — | 8 | 2 | 6 |
| 1.76 | — | 0.83 | — | 2 | 1 | 9 | 1.31 | — | 0.05 | — | 5 | 6 | 7 |
| 1.76 | — | 0.04 | — | 1 | 2 | 9 | 1.31 | — | 0.02 | — | 6 | 5 | 7 |
| 1.75 | — | 0.59 | — | 0 | 7 | 1 | 1.31 | — | 0.08 | — | 1 | 3 | 12 |
| 1.74 | — | 0.15 | — | 3 | 6 | 3 | 1.31 | — | 0.02 | — | 3 | 1 | 12 |
| 1.74 | — | 0.00 | — | 6 | 3 | 3 | 1.31 | — | 0.23 | — | 3 | 5 | 10 |
| 1.73 | 1.73 | 1.02 | 11.4 | 1 | 6 | 5 | 1.31 | — | 0.06 | — | 5 | 3 | 10 |
| 1.73 | — | 0.86 | — | 6 | 1 | 5 | 1.31 | — | 0.14 | — | 4 | 8 | 4 |
| 1.73 | — | 8.94 | — | 0 | 4 | 8 | 1.31 | — | 0.17 | — | 8 | 4 | 4 |
| 1.71 | 1.71 | 0.66 | 2.11 | 4 | 4 | 6 | 1.30 | — | 0.18 | — | 3 | 6 | 9 |
| 1.71 | — | 0.23 | — | 4 | 3 | 7 | 1.30 | — | 0.01 | — | 6 | 3 | 9 |
| 1.71 | — | 0.42 | — | 0 | 5 | 7 | 1.30 | — | 0.20 | — | 5 | 8 | 1 |
| 1.71 | — | 0.01 | — | 3 | 4 | 7 | 1.30 | — | 0.11 | — | 8 | 5 | 1 |
| 1.71 | — | 0.16 | — | 4 | 6 | 0 | 1.30 | — | 0.02 | — | 6 | 7 | 3 |
| 1.71 | — | 0.46 | — | 6 | 4 | 0 | 1.30 | — | 0.09 | — | 2 | 9 | 3 |
| 1.70 | — | 0.00 | — | 1 | 7 | 2 | 1.30 | — | 0.00 | — | 7 | 6 | 3 |
| 1.70 | — | 0.27 | — | 7 | 1 | 2 | 1.30 | — | 0.11 | — | 9 | 2 | 3 |
| 1.70 | — | 0.67 | — | 5 | 5 | 2 | 1.29 | — | 0.00 | — | 0 | 6 | 10 |
| 1.69 | — | 0.39 | — | 3 | 3 | 8 | 1.29 | — | 0.14 | — | 1 | 9 | 4 |
| 1.69 | — | 1.35 | — | 0 | 3 | 9 | 1.29 | — | 0.05 | — | 9 | 1 | 4 |
| 1.68 | — | 0.63 | — | 2 | 7 | 1 | 1.29 | — | 0.45 | — | 0 | 5 | 11 |
| 1.68 | — | 1.04 | — | 7 | 2 | 1 | 1.29 | — | 0.06 | — | 3 | 4 | 11 |
| 1.68 | — | 0.06 | — | 3 | 5 | 6 | 1.29 | — | 0.00 | — | 4 | 3 | 11 |
| 1.68 | — | 0.06 | — | 5 | 3 | 6 | 1.29 | — | 0.06 | — | 6 | 6 | 6 |
| 1.68 | — | 0.20 | — | 0 | 7 | 3 | 1.29 | — | 0.16 | — | 1 | 8 | 7 |
| 1.67 | 1.67 | 4.42 | 7.24 | 4 | 6 | 2 | 1.29 | — | 0.01 | — | 4 | 7 | 7 |
| 1.67 | — | 2.48 | — | 6 | 4 | 2 | 1.29 | — | 0.23 | — | 7 | 4 | 7 |
| 1.67 | 1.67 | 1.84 | 3.02 | 4 | 5 | 5 | 1.29 | — | 0.00 | — | 8 | 1 | 7 |
| 1.67 | — | 0.00 | — | 5 | 4 | 5 | 1.28 | — | 0.04 | — | 9 | 3 | 2 |
| 1.66 | — | 0.71 | — | 2 | 4 | 8 | 1.28 | — | 0.01 | — | 3 | 9 | 2 |
| 1.66 | — | 0.01 | — | 4 | 2 | 8 | 1.28 | — | 0.07 | — | 7 | 3 | 8 |
| 1.65 | 1.65 | 0.20 | 4.17 | 0 | 6 | 6 | 1.28 | — | 0.11 | — | 3 | 7 | 8 |
| 1.65 | — | 0.37 | — | 2 | 5 | 7 | 1.28 | — | 0.11 | — | 0 | 1 | 13 |

Continuation of Table 3

| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 |
|------|------|------|------|---|---|----|------|------|------|------|----|----|----|
| 1.65 | – | 3.04 | – | 5 | 2 | 7 | 1.28 | – | 0.08 | – | 0 | 7 | 9 |
| 1.64 | – | 0.57 | – | 1 | 1 | 10 | 1.27 | – | 0.12 | – | 5 | 7 | 6 |
| 1.63 | – | 1.10 | – | 2 | 3 | 9 | 1.27 | – | 0.10 | – | 7 | 5 | 6 |
| 1.63 | – | 1.08 | – | 3 | 2 | 9 | 1.27 | – | 0.33 | – | 5 | 8 | 3 |
| 1.62 | – | 0.04 | – | 2 | 7 | 3 | 1.27 | – | 0.04 | – | 8 | 5 | 3 |
| 1.62 | – | 0.00 | – | 7 | 2 | 3 | 1.27 | – | 0.16 | – | 0 | 4 | 12 |
| 1.61 | 1.61 | 1.37 | 4.17 | 0 | 2 | 10 | 1.27 | – | 0.38 | – | 2 | 6 | 10 |
| 1.61 | – | 0.44 | – | 3 | 6 | 5 | 1.27 | – | 0.54 | – | 6 | 2 | 10 |
| 1.61 | – | 0.02 | – | 6 | 3 | 5 | 1.26 | – | 0.11 | – | 0 | 9 | 5 |
| 1.61 | – | 0.30 | – | 1 | 7 | 4 | 1.26 | – | 0.00 | – | 2 | 5 | 11 |
| 1.61 | – | 0.04 | – | 7 | 1 | 4 | 1.26 | – | 0.36 | – | 5 | 2 | 11 |
| 1.61 | – | 0.03 | – | 5 | 5 | 4 | 1.58 | – | 1.43 | – | 4 | 1 | 9 |
| 1.60 | – | 0.39 | – | 2 | 6 | 6 | 1.57 | – | 1.12 | – | 5 | 6 | 1 |
| 1.60 | – | 0.88 | – | 6 | 2 | 6 | 1.57 | – | 0.09 | – | 6 | 5 | 1 |
| 1.59 | – | 0.00 | – | 3 | 7 | 2 | 1.56 | – | 1.07 | – | 2 | 2 | 10 |
| 1.59 | – | 0.00 | – | 7 | 3 | 2 | 1.56 | – | 0.21 | – | 0 | 7 | 5 |
| 1.58 | 1.58 | 0.04 | 1.07 | 4 | 6 | 4 | 1.54 | 1.54 | 0.38 | 6.21 | 1 | 6 | 7 |
| 1.58 | – | 0.01 | – | 6 | 4 | 4 | 1.54 | – | 0.01 | – | 6 | 1 | 7 |
| 1.58 | – | 0.00 | – | 5 | 1 | 8 | 1.54 | – | 5.94 | – | 0 | 8 | 0 |
| 1.58 | – | 0.02 | – | 1 | 5 | 8 | 1.53 | – | 0.08 | – | 1 | 3 | 10 |
| 1.58 | – | 0.34 | – | 1 | 4 | 9 | 1.53 | – | 0.00 | – | 3 | 1 | 10 |
| 1.58 | – | 1.43 | – | 4 | 1 | 9 | 1.52 | 1.52 | 0.22 | 1.05 | 1 | 8 | 1 |

The disagreement factor is $R=9.8845\%$.

Table 4 shows the interatomic distances of the K_3VF_6 compound under the number 00-037-0738 in the pdf-2 database for 2004.

Table 4

Interatomic distances of the test compound

| Atom1 | Atom2 | Distance, Å° | Atom1 | Atom2 | Distance, Å° | Atom1 | Atom2 | Distance, Å° |
|-------|-------|--------------|-------|-------|--------------|-------|-------|--------------|
| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
| K1 | K5 | 1.677 | – | F7 | 3.364 | V2 | F5 | 2.045 |
| – | F1 | 1.830 | – | K5 | 3.437 | – | F7 | 2.534 |
| – | F4 | 2.128 | – | F1 | 3.479 | – | F4 | 2.590 |
| – | F3 | 2.210 | – | F7 | 3.482 | – | F1 | 2.827 |
| – | K3 | 2.298 | – | F3 | 2.899 | – | K5 | 2.883 |
| – | F7 | 2.536 | – | F1 | 2.961 | – | F3 | 2.969 |
| – | F6 | 2.884 | – | F5 | 3.028 | – | F8 | 3.067 |
| – | F8 | 2.972 | – | F6 | 3.030 | F4 | F7 | 1.946 |
| – | F5 | 3.326 | – | F7 | 3.329 | – | K1 | 2.128 |
| – | F6 | 3.347 | – | F4 | 3.400 | – | F6 | 2.326 |
| – | V3 | 3.424 | – | F4 | 3.452 | – | K2 | 2.390 |
| K5 | K1 | 1.677 | – | K3 | 3.475 | – | V2 | 2.590 |
| – | F6 | 2.070 | – | F5 | 3.497 | – | K5 | 2.710 |

Continuation of Table 4

| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
|----|----|-------|----|----|-------|----|----|-------|
| – | F8 | 2.462 | V3 | F8 | 1.073 | – | K5 | 2.767 |
| – | K3 | 2.617 | – | K5 | 3.117 | – | F2 | 2.819 |
| – | F1 | 2.682 | – | K1 | 3.424 | – | F5 | 3.000 |
| – | F2 | 2.687 | V4 | F5 | 2.187 | – | F3 | 3.069 |
| – | F4 | 2.710 | – | K3 | 2.433 | – | K3 | 3.400 |
| – | F4 | 2.767 | – | F7 | 2.744 | – | K3 | 3.452 |
| – | V2 | 2.883 | – | F3 | 3.123 | – | F6 | 3.482 |
| – | K2 | 2.992 | F5 | F7 | 1.577 | F6 | K2 | 1.983 |
| – | V3 | 3.117 | – | V2 | 2.045 | – | K5 | 2.070 |
| – | F7 | 3.228 | – | V4 | 2.187 | – | K2 | 2.307 |
| – | K2 | 3.437 | – | F3 | 2.190 | – | F1 | 2.311 |
| F1 | K1 | 1.830 | – | F4 | 3.000 | – | F4 | 2.326 |
| – | F6 | 2.311 | – | K3 | 3.028 | – | F3 | 2.470 |
| – | K2 | 2.380 | – | K1 | 3.326 | – | F7 | 2.528 |
| – | F2 | 2.636 | – | K3 | 3.497 | – | K1 | 2.884 |
| – | K5 | 2.682 | F2 | F1 | 2.636 | – | K3 | 3.030 |
| – | F3 | 2.738 | – | K5 | 2.687 | – | F2 | 3.146 |
| – | V2 | 2.827 | – | F4 | 2.819 | – | K1 | 3.347 |
| – | K3 | 2.961 | – | F6 | 3.146 | – | F4 | 3.482 |
| – | F8 | 3.003 | – | F8 | 3.252 | F7 | F5 | 1.577 |
| – | F3 | 3.405 | F3 | F5 | 2.190 | – | F4 | 1.946 |
| – | K2 | 3.479 | – | K1 | 2.210 | – | K3 | 2.322 |
| K2 | F6 | 1.983 | – | F6 | 2.470 | – | F6 | 2.528 |
| – | F6 | 2.307 | – | F7 | 2.663 | – | V2 | 2.534 |
| – | F1 | 2.380 | – | F7 | 2.732 | – | K1 | 2.536 |
| – | F4 | 2.390 | – | F1 | 2.738 | – | F3 | 2.663 |
| – | K3 | 2.471 | – | K2 | 2.868 | – | F3 | 2.732 |
| – | F3 | 2.868 | – | K3 | 2.892 | – | V4 | 2.744 |
| – | K5 | 2.992 | – | F3 | 2.899 | – | F7 | 3.151 |
| – | F3 | 2.998 | – | F1 | 2.961 | – | K5 | 3.228 |
| – | F7 | 3.364 | – | F5 | 3.028 | – | K3 | 3.329 |
| – | K5 | 3.437 | – | F6 | 3.030 | – | K2 | 3.364 |
| – | F1 | 3.479 | – | F7 | 3.329 | – | K2 | 3.482 |
| – | F7 | 3.482 | – | F4 | 3.400 | F8 | V3 | 1.073 |
| K3 | K1 | 2.298 | – | K3 | 2.899 | – | F8 | 2.147 |
| – | F7 | 2.322 | – | V2 | 2.969 | – | K5 | 2.462 |
| – | V4 | 2.433 | – | K2 | 2.998 | – | K1 | 2.972 |
| – | K2 | 2.471 | – | F4 | 3.069 | – | F1 | 3.003 |
| – | K5 | 2.617 | – | V4 | 3.123 | – | V2 | 3.067 |
| – | F3 | 2.892 | – | F1 | 3.405 | – | F2 | 3.252 |

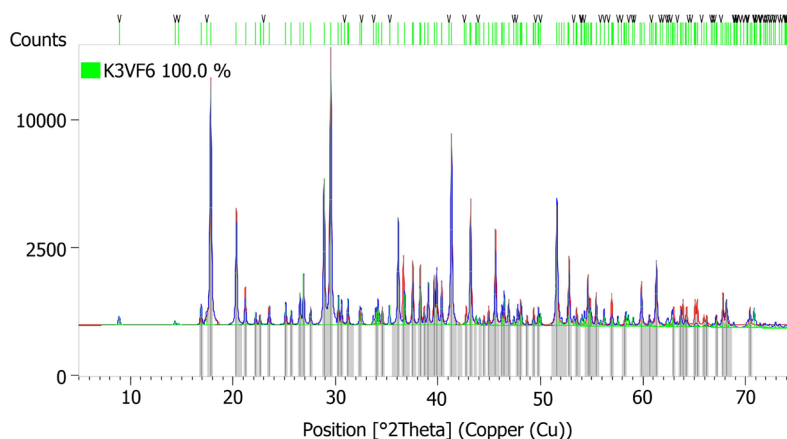


Fig. 2. The resulting diffraction patterns

Fig. 2 shows the resulting diffractograms calculated from the proposed microstructural parameters of the K_3VF_6 compound

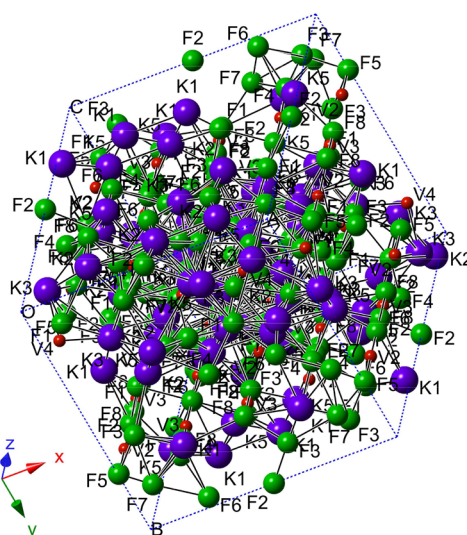


Fig. 3. Crystal structure image of K_3VF_6 compound

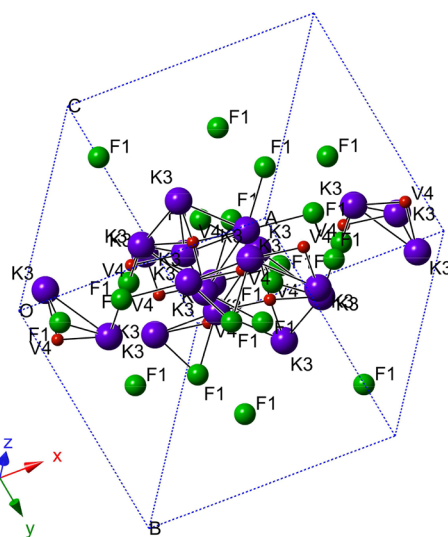


Fig. 4. Image fragment of the crystal structure of K_3VF_6 compound

Fig. 3 shows the crystal structure of the K_3VF_6 tetragonal syngony of the space group I 41/a, belonging to its own type of structure. As can be seen from **Fig. 4**, the V atoms form triangular-shaped bonds with fluorine and potassium atoms.

4. Discussion of results

As follows from the literature review, the test compound has a simple state diagram of the KCl-KF- K_3VF_6 system, which means that other compounds are not formed during the K_3VF_6 synthesis. Various methods of synthesis of this compound make it possible to obtain the test compound in its pure form, and the diffraction patterns are completely consistent with the single-phase structure of this substance.

In addition, the literature indicates the existence of three polymorphic modifications of this compound [8], such as alpha, beta, and gamma phases.

The crystal structure of the β phase is studied in [13], and belongs to the $[NH_4]_3FeF_6$ structural type.

The crystal structure of the α phase is studied in this work and assigned to the intrinsic structural type of tetragonal syngony. So, at temperatures between 298 K and 453 K (for which the captured diffractograms [8] also exist), there is a polymorphic transformation from a tetragonal lattice to a cubic one.

The interatomic distances calculated for the proposed structural model of the K_3VF_6 α phase indicate the reliability of the existence of this structural model.

From the fragment of the structure in **Fig. 4**, it follows that the vanadium atoms form a tri-cycle bond with the potassium and fluorine atoms.

5. Conclusions

The X-ray phase analysis does not reveal the presence in the test compound of several phases, are presented in **Table 1**. So let's conclude that the connection is single-phase.

Using the HighScorePlus Treor program module, X-ray diffraction patterns of the K_3VF_6 compound are indexed under the number 00-037-0738 in the PDF-2 database for 2004.

The diffraction spectrum of the compound is indexed in tetragonal system with lattice periods $a=5.929$ Å; $b=8.294$ Å; $c=5.914$ Å. The space group of symmetry I 41/a is possible.

Using the HighScorePlus 3.0 program, the Rietveld method is used to study the diffraction spectrum of the compound under the number 00-037-0738. The structural model is proposed: the spatial symmetry group I 41/a (88), with lattice periods $a=12.3524(4)$ Å; $b=12.3524(4)$ Å; $c=16.7447(7)$ Å.

The disagreement factor is $R=9.88451$ %. The microstructural parameters are given in **Table 2**.

The crystal structure of this compound may belong to a new type of structure.

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